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Eri Silk Fibroin Derived Carbon Quantum Dots for Photocatalytic Dye Degradation

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Presentation Outline

01 Introduction

02 Objectives

03 Synthesis of CQD@ESF

04 Results and discussion

05 Summary and conclusions

06 References

Introduction

- Rapid industrialization has led to severe wastewater pollution

- Major contaminants:
  - Heavy metal ions
  - Dyes
  - Pharmaceuticals
  - Organic pollutants

- Conventional treatment methods suffer from:
  - Incomplete removal
  - Secondary pollution
  - High energy demand



Fig. 1: water pollution

- Semiconductor photocatalysis has emerged as a promising green technology for wastewater remediation

Carbon Quantum Dots

- New class of fluorescence carbon nanoparticles composed of discrete quasi-spherical carbonaceous material with a particle size of less than 10 nm (Yan et al., 2020)
- Commonly called carbon nano dots (CNDs) or simply carbon dots (CDs)
- CQDs were first discovered by Xu et al., in 2004 accidentally during the purification of single wall carbon nanotubes

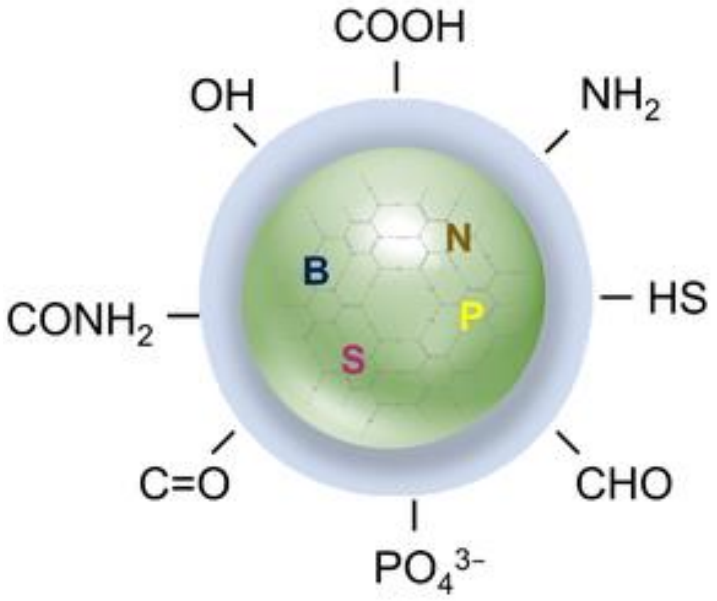


Fig. 2:Generalized structure of CQDs

Carbon Quantum Dots

Composition and structure

- Core is mostly carbon
- Contains 5-50 wt% oxygen and other elements (H, N, P & S)
- Morphology is mostly quasi-spherical and structure can be graphitic or amorphous

Properties

- Strong photoluminescence
- High water solubility
- Biocompatibility and Low toxicity
- Efficient Photocatalysts
- Environment friendly

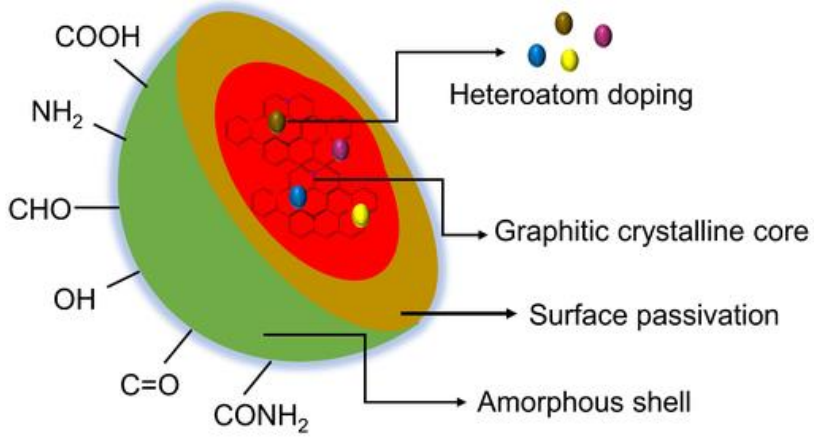


Fig. 3: Illustration of the core–shell structure of CQDs

Eri silk

- **Regional availability:** Abundantly available in Assam and Northeast India.
- **Renewable resource:** Naturally derived, protein-based biopolymer.
- **Rich protein composition:** Silk fibroin contains carbon, nitrogen and oxygen.
- **Nitrogen-containing precursor:** Offers the potential for nitrogen-containing surface functionalities.
- **Sustainable approach:** Supports the utilization of natural biomaterials for nanomaterial synthesis.

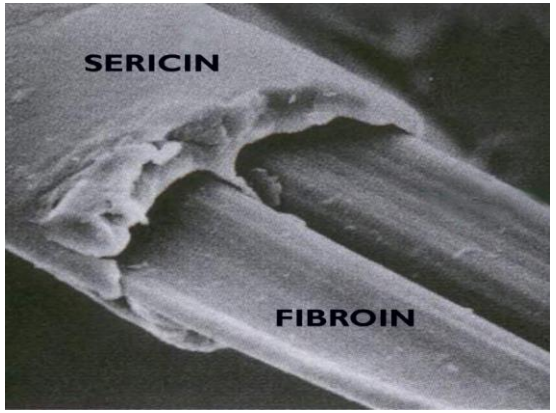


Fig.4. Fibroin and Sericin interlinked together. (Zhengzhong Shao et al., Shao, Z., Vollrath, F. Surprising strength of silkworm silk. Nature 418, 741 (2002))

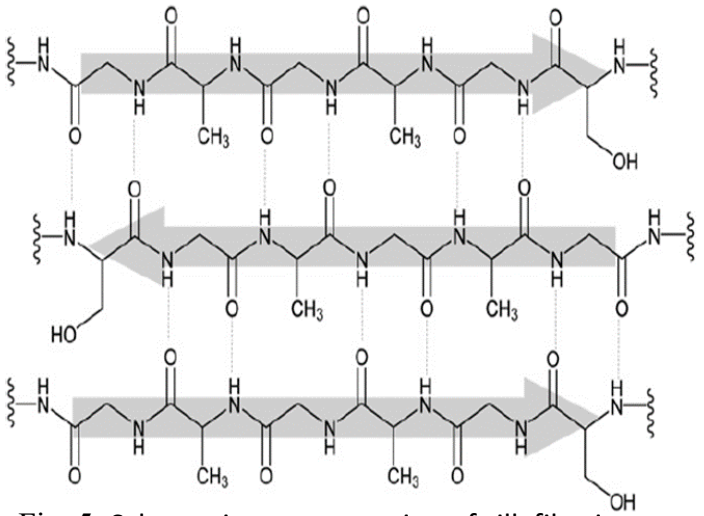


Fig. 5: Schematic representation of silk fibroin polypeptide structure (J. Mater. Chem.,)

Mechanism of photocatalysis

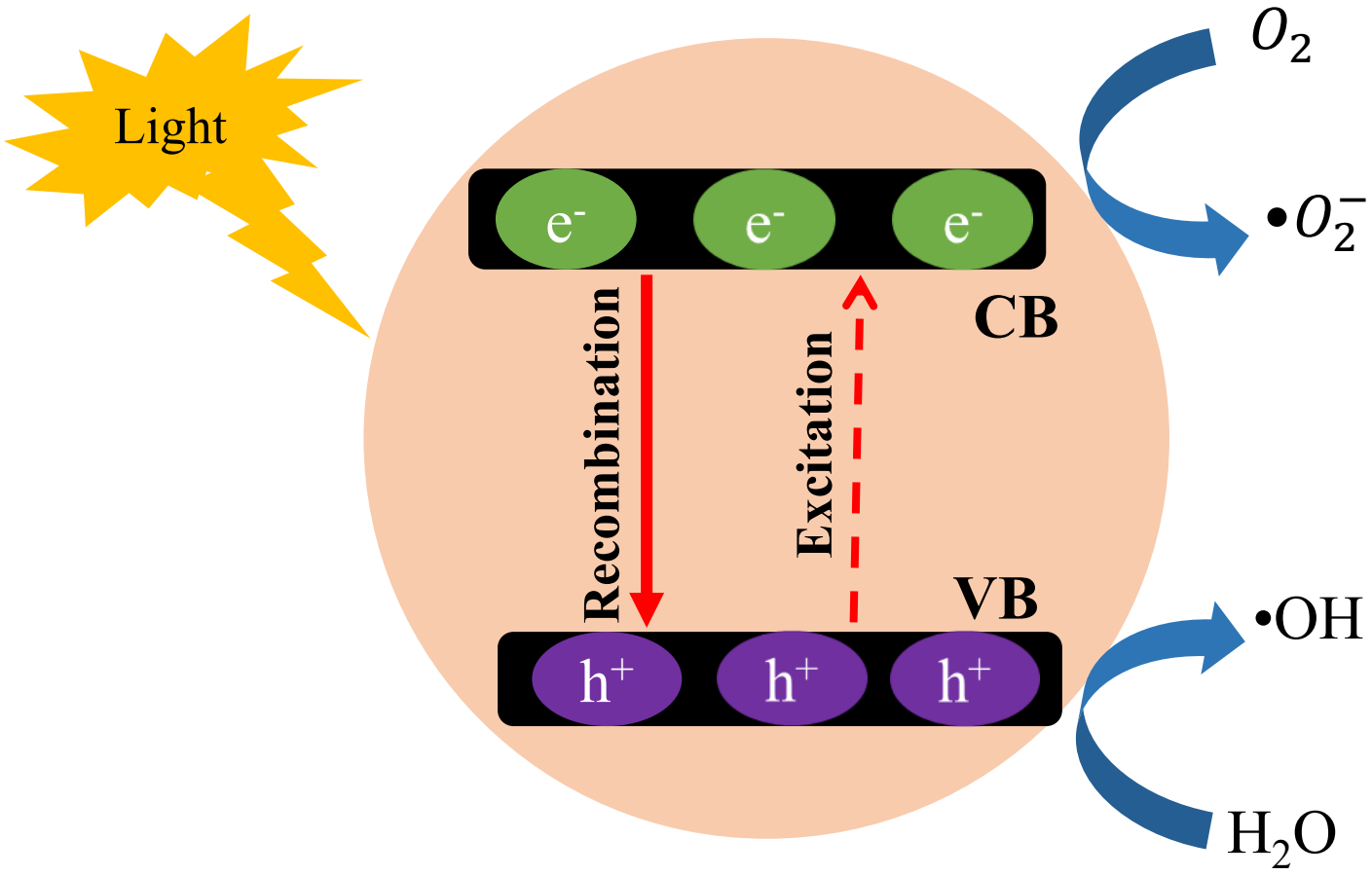


Fig. 6: Mechanism of photocatalysis

Role of CQD in Photocatalysis

- ❑ Enhances visible-light absorption
- ❑ Improves charge separation
- ❑ Reduces recombination
- ❑ Increases photocatalytic efficiency

Objectives

1. To synthesize CQDs from Eri silk fibroin (ESF) using the hydrothermal method at different temperatures (120, 135 and 150 °C).
2. To characterize the synthesized CQDs using UV–Vis, FTIR and XRD.
3. To evaluate their photocatalytic performance towards the degradation of methylene blue and congo red dye.

Synthesis of CQD@ESF

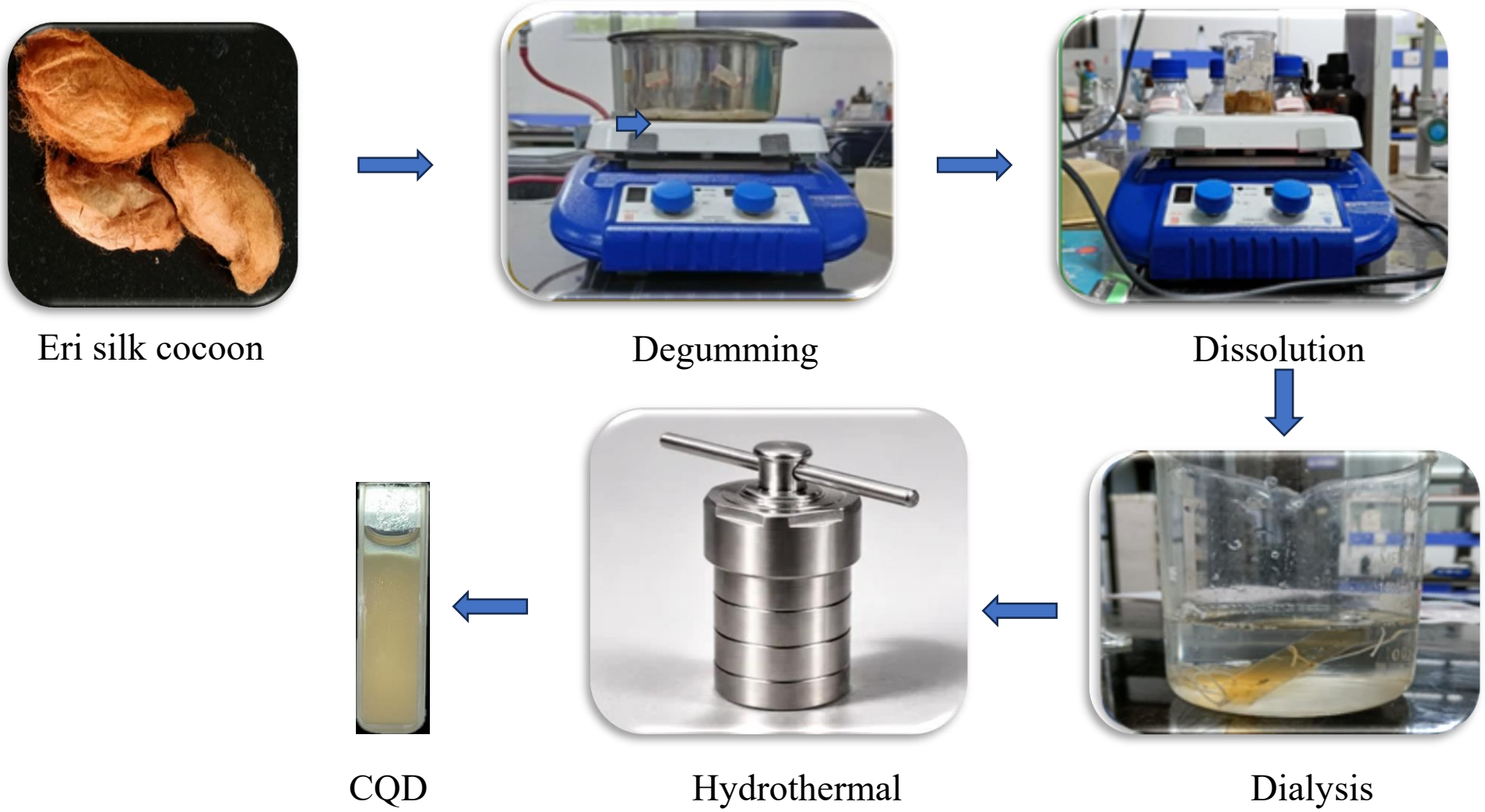


Fig. 7: Synthesis of ESF derived CQDs

Photocatalytic experiment



Photochemical reactor



Fig. 8: Photocatalytic experimental setup

Results and Discussions

UV-Visible spectrophotometer (UV-Vis)

- All three samples exhibit strong absorption in the UV region (approximately 200–230 nm), indicating electronic transitions($\pi \rightarrow \pi^*$) associated with conjugated carbon domains.
- A broad shoulder is observed around 270–285 nm, which may be attributed to n–π^* transitions involving surface functional groups such as C=O.
- The gradual absorption tail extending from the UV into the visible region suggests the presence of surface/defect-related electronic states.
- The higher absorbance of the 150 °C sample may indicate differences in its carbonization, conjugation or surface-state characteristics.

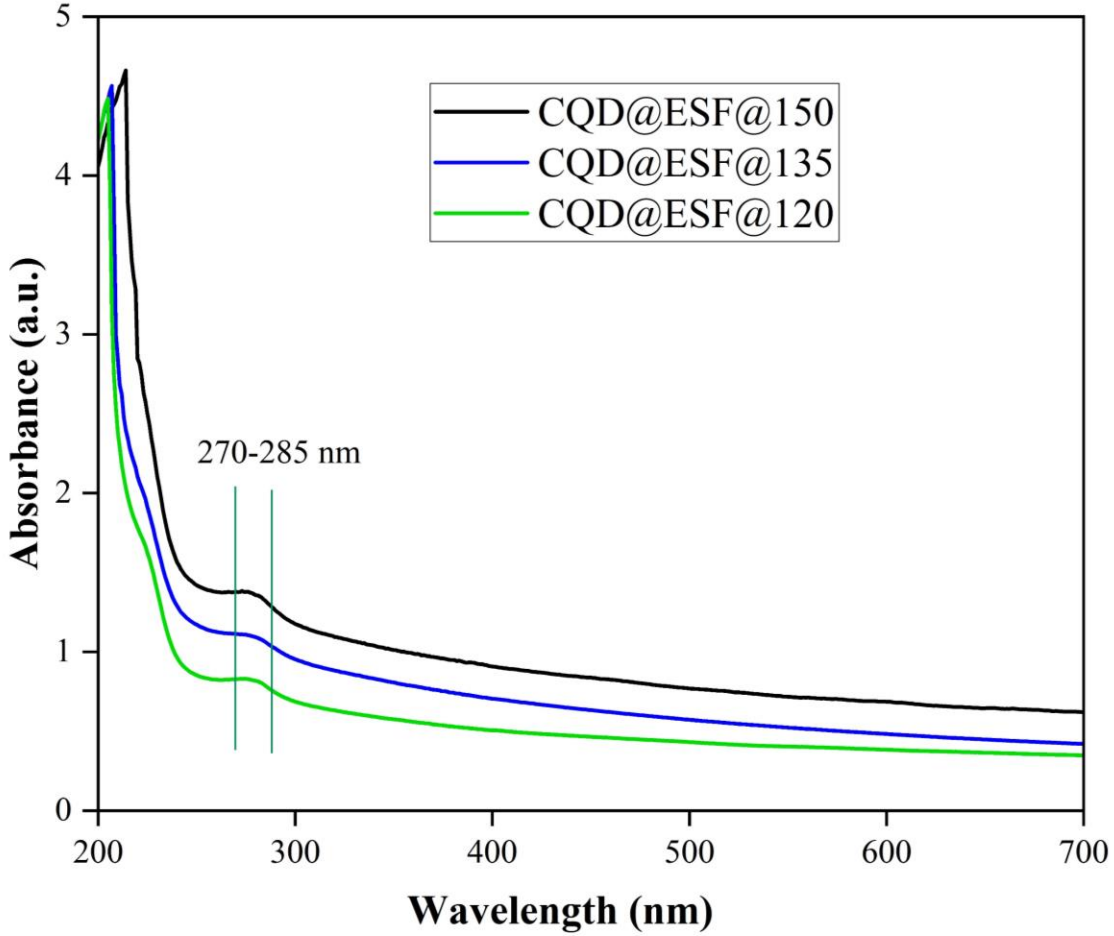


Fig 9: UV-Vis spectra of CQD@ESF

Fourier Transform Infrared Spectroscopy (FTIR)

- Broad absorption in the 3200–3600 cm^{-1} region indicates O–H/N–H stretching vibrations *(Li et al., 2012)*.
- Bands around 2920–2960 cm^{-1} are associated with aliphatic C–H stretching.
- The amide I band (~1630–1660 cm^{-1}) is associated with carbonyl stretching.
- The amide II band (~1510–1540 cm^{-1}) corresponds to N–H bending and C–N stretching.
- Amide III vibrations are observed in the 1200–1300 cm^{-1} region.
- The spectral features suggest the presence of protein-derived functional groups on the CQD@ESF samples.

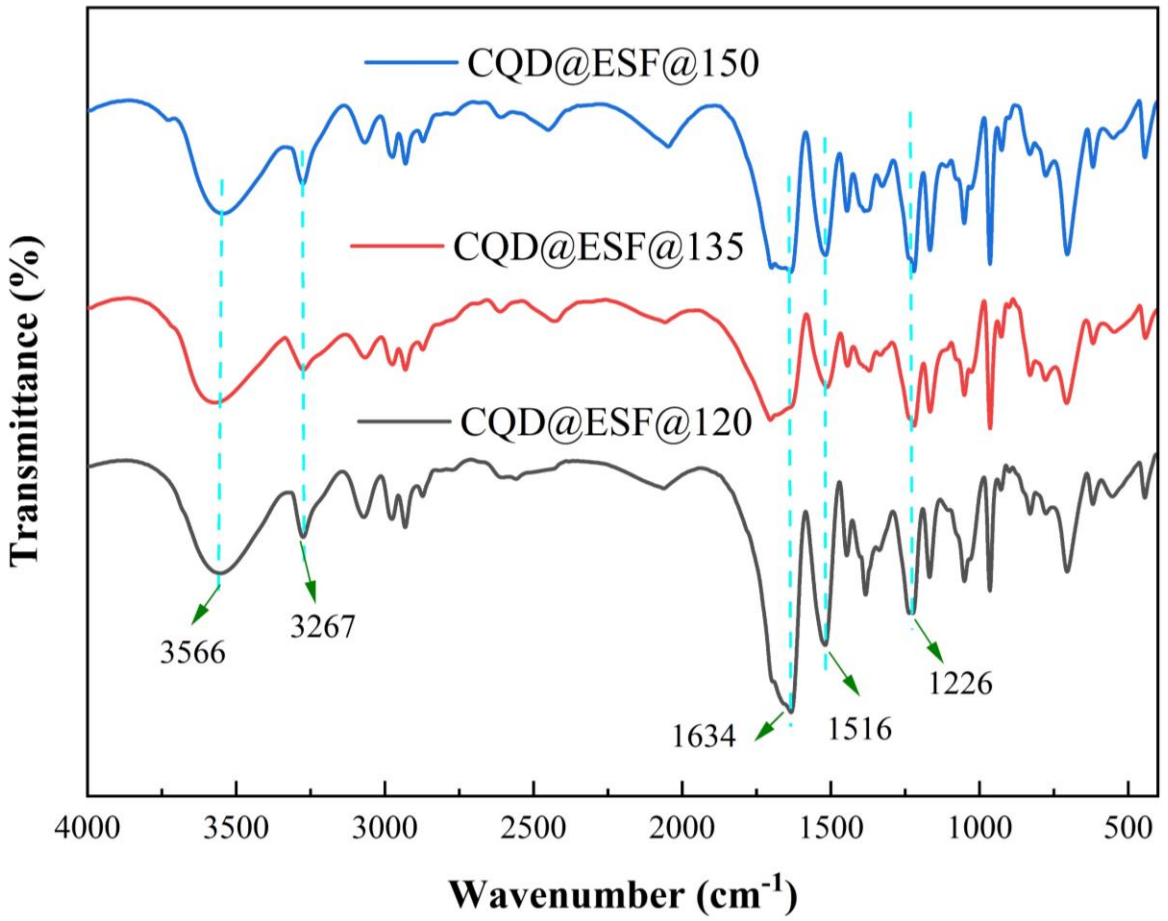


Fig 10: FTIR of CQD@ESF

X-Ray Diffractometer (XRD)

- A broad diffraction maximum around 22° is attributed to the (002) plane of poorly ordered carbon *(JCPDS No. 41-1487)*.
- A weak broad feature around 42–43° may be assigned to the (100) plane, associated with in-plane ordering of carbon domains.
- The broad nature of both features indicates limited long-range structural order.
- The differences among the samples suggest possible temperature-dependent changes in carbon structural organization.

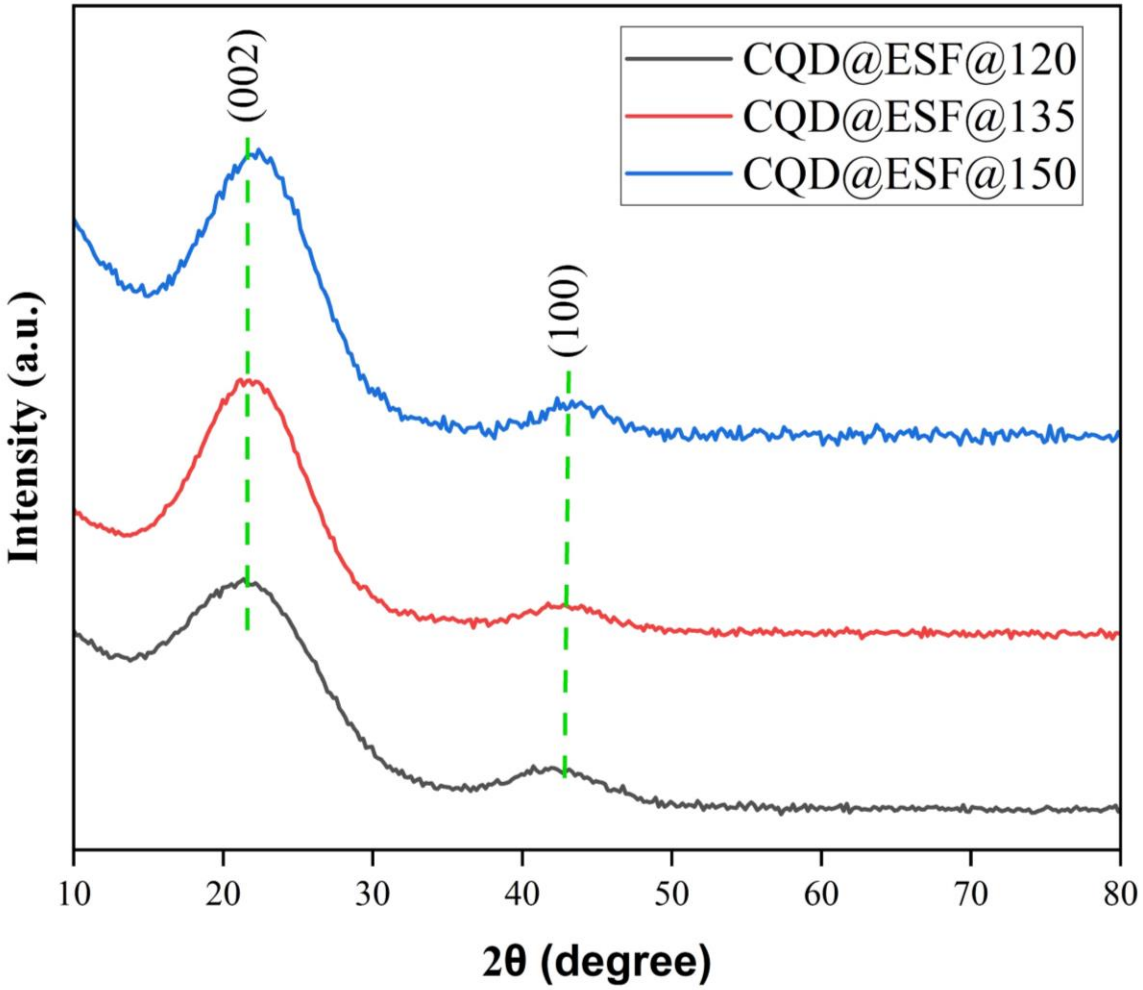
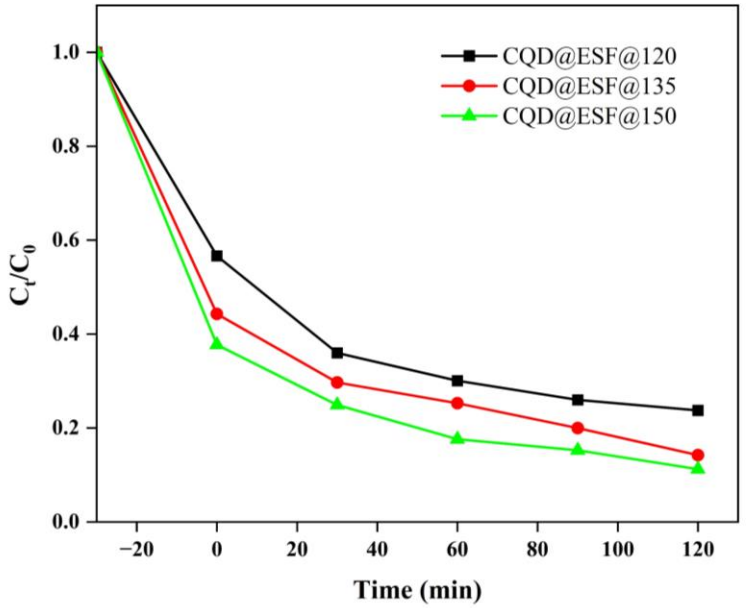


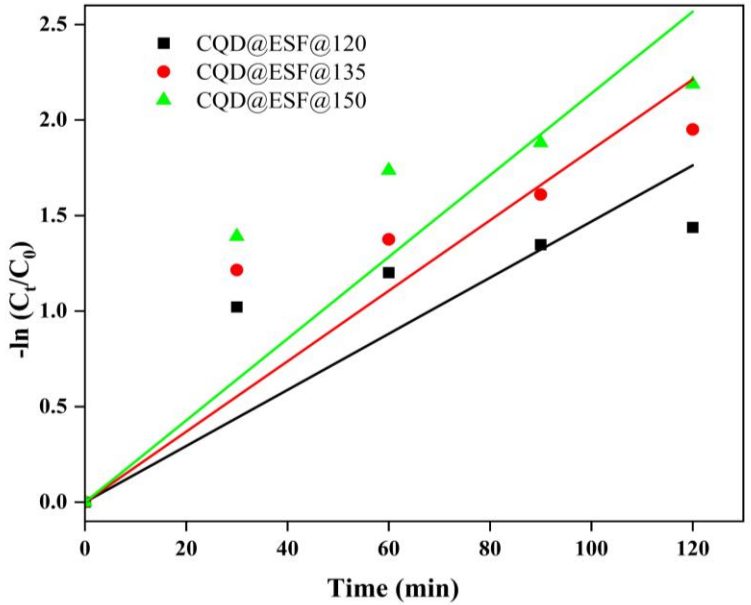
Fig 11: XRD spectra of CQD@ESF

Photocatalytic Degradation of Methylene Blue Dye

a)



b)



Degradation efficiency(η):

$$(1 - \frac{C_t}{C_0}) \times 100\%$$

Where, C_0→initial concentration of dye, C_t→ concentration after time t and rate constant(k):

$$-\ln\frac{C_t}{C_0} = k \times t$$

(Li et al. 2020)

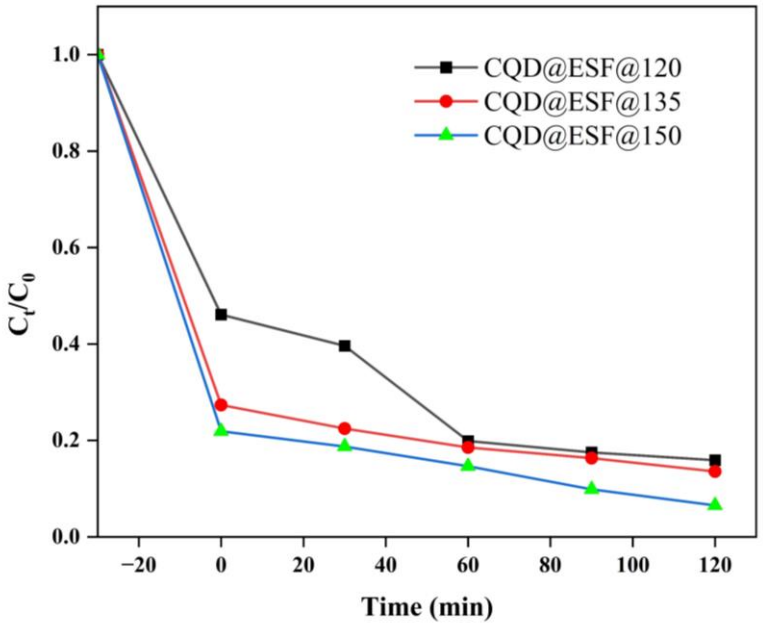
Fig 12: (a) Photocatalytic degradation of MB dye (b) rate constant

Samples	Photocatalytic degradation efficiency under visible light (%)	Rate constant (10^{-3}) (min^{-1})
CQD@ESF@120	76.25	14.69
CQD@ESF@135	85.77	18.44
CQD@ESF@150	88.77	21.39

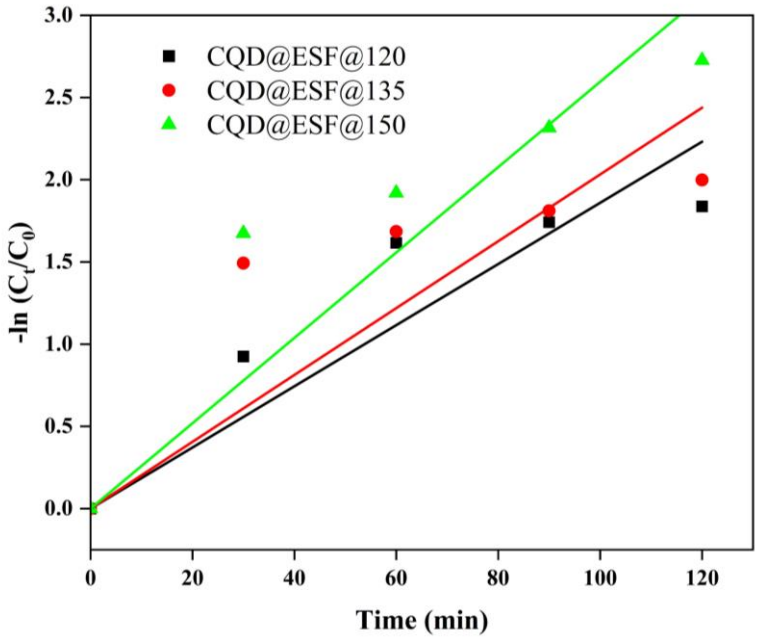
- Photocatalytic performance improves with increasing hydrothermal synthesis temperature.
- CQD@ESF@150 exhibits the highest degradation efficiency (~89%) after 120 min.
- The pseudo-first-order kinetic model indicates an increasing apparent rate constant with synthesis temperature.

Photocatalytic Degradation of Congo Red Dye

a)



b)



Degradation efficiency(η):

$$(1 - \frac{C_t}{C_0}) \times 100\%$$

Where, C_0→initial concentration of dye, C_t→ concentration after time t and rate constant(k):

$$-\ln\frac{C_t}{C_0} = k \times t$$

(Li et al. 2020)

Fig 13: (a) Photocatalytic degradation of CR dye (b) rate constant

Samples	Photocatalytic degradation efficiency under visible light (%)	Rate constant (10^{-3}) (min^{-1})
CQD@ESF@120	84	18.59
CQD@ESF@135	86.5	20.32
CQD@ESF@150	93	25.96

- CQD@ESF@150 exhibits the highest degradation efficiency (~93%) after 120 min.
- The enhanced performance of CQD@ESF@150 may be associated with temperature-dependent changes in the optical and surface properties of CQD@ESF..

Summary and conclusions

- ESF-derived CQDs were successfully prepared using the hydrothermal approach at 120, 135 and 150 °C.
- UV–Vis analysis revealed strong UV absorption, with possible $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions.
- FTIR confirmed the presence of characteristic protein-derived functional groups.
- XRD revealed broad diffraction features, suggesting poorly ordered carbon structures.
- All three samples demonstrated photocatalytic activity towards MB and CR under visible light irradiation.
- The 150 °C sample (CQD@ESF@150) exhibited the highest dye degradation efficiencies.

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Thank You